PATENT SPECIFICATION (11) 1219946

1219946

DRAWINGS ATTACHED

(21) Application No. 38926/68 (22) Filed 14 Aug. 1968

(23) Complete Specification filed 15 July 1969

(45) Complete Specification published 20 Jan. 1971

(51) International Classification B 29 f 3/02

(52) Index at acceptance

B5A 1G10 1G5A 1G5E 2D1X 2L
B1C 1 14 19G4A2 19G4B 19GU 9

(72) Inventor PETER JOHN BARKER

(54) EXTRUSION PROCESS AND APPARATUS

(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London S.W.1., a British Company,

do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an extrusion process and to apparatus therefor, and in particular to a process and apparatus for incorporating additives into a thermoplastic polymeric material during the extrusion thereof.

Conventional processes for the incorporation of additives such as pigments, fillers, cross-linked polymers, and dyestuffs into thermoplastic polymeric materials consist of mixing the additive with the raw polymeric material by, for example, tumble blending, and then to mill and/or extrude the resulting mixture to obtain a homogeneous mixture with the additive evenly dispersed throughout the polymeric material.

It would often be desirable to introduce the additive into the polymeric material in such a way that the quantity of additive incorporated can be controlled readily. In British Patent Specification 1,049,773 it has been proposed to achieve this object by injecting a dispersion of the additive in a volatile diluent into the barrel of a vented screw extruder during the extrusion of the polymeric material and to remove the volatile diluent through a vacuum extraction or devolatilisation zone of the extruder. However, such a process involves tapping the extruder barrel before the devolatilisation zone in order to introduce the additive dispersion and this often involves substantial engineering complications as the clearances between the extruder screw and the barrel are generally very small. Also there is sometimes a tendency for the additive to settle out in the additive dispersion giving rise to blockages or to uneven distribution of the additive in the polymeric material.

We have devised a process whereby these difficulties may be overcome.

According to the present invention we provide an extrusion process in which a molten mass of a thermoplastic polymeric material (A) is extruded utilising screw extrusion apparatus having at least two zones through which the polymeric material (A) is conveyed by means of a screw or screws and a duct connecting said zones so that the thermoplastic polymeric material (A) can pass from one zone to the other, characterised in that an intimate, mixture of an additive and a thermoplastic polymeric material (B) is injected into said duct in the form of a melt while the molten thermoplastic polymeric material (A) is passing therethrough.

Apparatus suitable for use in the above process may comprise a screw extrusion apparatus having at least two zones through which a polymeric material (A) may be conveyed by means of a screw and a duct connecting said zones so that the polymeric material (A) can pass from one zone to the other, and injection means for injecting a molten mixture of an additive and a thermoplastic polymeric material (B) into said duct.

In one form the apparatus may consist of at least two separate screw extruders, one feeding directly into the feed point of the other. For example a twin screw extruder may be arranged to extrude the polymeric material (A) into the barrel of a single screw extruder via a short unstirred duct. It is into that duct that the mixture of the additive and polymeric material (B) is injected.

An alternative system is to use an extruder having two zones separated by a cylindrical member fastened to the extruder screw and rotatable therewith which member acts as a plug to prevent flow of the polymeric material (A) along the extruder barrel from one zone to the other and having a duct passing along the barrel wall connecting the zones, thereby bypassing the cylindrical member. An example of this type of apparatus is described in our British Patent Specification 927,501 although it is not essential that a valve is incorporated in the bypass duct in the present invention. The apparatus described in that specification

[*Price 5s. 0d. (25p)*]

may be fitted with any suitable means for injecting the mixture of the thermoplastic polymeric material (B) and the additive into the duct. The means for injecting the mixture of polymeric material (B) and the additive into the duct preferably consists of a small ram or screw extruder, particularly the latter.

The mixture of additive and polymeric material (B) may be made by mixing the constituents in powder form, for example by tumble blending or it may be a masterbatch prepared by melt compounding, for example milling, the polymeric material (B) with the additive. This mixture is injected as a melt into the thermoplastic polymeric material (A) in the duct.

Where the mixture of the additive and the polymeric material (B) is injected into the duct using an extruder as the injection means, often there is no need to separately heat this secondary extruder as sufficient heat to fuse the polymeric material (B) to form the melt will generally be conducted from the bypass of the main extruder.

In order to obtain the most even dispersion of the additive in the final polymer composition, the additive/polymeric material (B) mixture is preferably not injected into the duct in a direction normal to the flow of the polymeric material (A) through the duct, but is preferably injected in a direction substantially in line with, or even more preferably, in a direction substantially counter to the flow of the polymeric material (A) through the duct.

As in the case of utilising an extruder of the type depicted in British Patent Specification 927,501, the extruder is preferably fitted with a devolatilisation zone which conveniently takes the form of a low compression zone, into which the polymeric material (A) containing the additive/polymeric material (B) mixture is passed on leaving the duct, and a port or vent in the extruder barrel wall communicating with the low compression zone, to which port or vent suction is applied.

If desired the injection means used for introducing the mixture of the additive and polymeric material (B) into the duct may also be arranged to provide a valve to regulate the flow of polymeric material (A) through the duct.

In order to improve the dispersion of the additive in the polymeric material (A), particularly when using polymeric materials that give very viscous melts, we prefer to incorporate a mixing head in a zone of the extrusion apparatus situated after introduction of the mixture of the additive and the polymeric material (B). Examples of suitable mixing heads are those described in our United Kingdom Patent Specifications 787,764 and 843,849.

The polymeric materials (A) and (B) may be the same or different. Where they are different polymeric material (B) may itself be the additive, for example an impact modifier, which is desired to be dispersed in polymeric material (A).

However, preferably polymeric materials (A) and (B) are the same or similar materials. Examples of suitable polymeric materials include polyolefines such as polyethylene, polypropylene and poly - 4 - methyl pentene - 1, vinyl polymers such as polystyrene, and vinyl chloride polymers and copolymers, vinylidene chloride polymers and copolymers, acrylic polymers such as polymethyl methacrylate, polyamides and polyoxymethylenes.

The invention is particularly suitable for use in the production of polymethyl methacrylate compositions containing additives by the extrusion polymerisation process described in our British Patent Specification 875,853 wherein monomeric methyl methacrylate or a partially polymerised methyl methacrylate syrup is fed to a screw extruder together with a particular catalyst and is polymerised therein at an elevated temperature. In such a process an extruder fitted with a devolatilisation zone is often utilised to remove any unreacted monomeric methyl methacrylate and the additive in intimate admixture with already polymerised polymethyl methacrylate may be injected into the bypass of the extruder just before the devolatilisation zone.

Examples of suitable additives include pigments; fillers, for example glass fibres or mica; stabilisers; lubricants; crosslinked polymers; and dyestuffs.

The invention is illustrated by the following Examples in which all parts and percentages are expressed by weight.

Example 1

100 parts of methyl methacrylate, 0.1 part ditertiary butyl peroxide and 0.1 part lauryl mercaptan were pumped at a pressure of 100 p.s.i. into the feed pocket of a twin-screw extruder. The extruder screws were 3½" diameter, their centres being 3" apart. The extruder was 49" long and had 4 heating zones (1) 9"—18" from the feed pocket (2) 18"—27" from the feed pocket (3) 27"—36" from the feed pocket and (4) 36"—49" from the feed pocket. The heating zones were set at (1) 160°C, (2) 155°C, (3) 155°C and (4) 175°C. The screws were rotated at 7 revs./min. The outlet from the twin-screw extruder was connected to the feed pocket of a 1½" single screw extruder fitted with two vacuum extraction zones. This single screw extruder is depicted in Figure 1 the drawing of which accompanying the Provisional Specification which is a longitudinal part elevation part section of the extruder.

The extruder has a screw having high compression zones 1, 2, immediately followed by cylindrical members 3, 4 effectively blanking off the high compression zones 1, 2, from the following zones 5, 6 of the extruder. Zones

5 and 6 are low compression zones to which suction is applied via ports 7, 8. Ducts 9, 10 are provided in the extruder barrel wall communicating zone 1 with zone 5 and zone 2 with zone 6 so that polymer in zone 1 can bypass the cylindrical member 3 and pass into zone 5 and likewise polymer in zone 2 can bypass the cylindrical member 4 and pass into zone 6.

A ½" single screw extruder 11 is provided to inject a melt compounded mixture of 50 parts of barium sulphate pigment and 50 parts of a polymethyl methacrylate, having a reduced viscosity of 0.5 dl./g. as measured on a solution of 1 g. of the polymer in 100 ml. of chloroform at 20°C, as a melt into the duct 10 of the second vacuum extraction section against the flow of the polymer through duct 10. This arrangement is shown more clearly in Figure 2 the drawing of which accompanies the provisional specification which is part of a longitudinal cross-section of the apparatus in the vicinity of the second bypass duct 10.

The pigment/polymethyl methacrylate mixture was injected at a rate of 0.8 lb./hr. In the devolatilisation zones 5 and 6, excess of unreacted monomer is removed and the resultant polymethyl methacrylate containing the pigment evenly dispersed therein is then extruded at an output rate of 20 lb./hr. through a 12 inch slit die 12 after passing through a further high compression zone 13, to give an opal sheet with excellent pigment dispersion.

EXAMPLE 2

Polymethyl methacrylate was extruded at 200 lb./hr. through a 4½" single screw extruder fitted with a bypass and vacuum extraction system of the type depicted in Figure 1 and also fitted with a pyramoidal mixing head of the type described in our United Kingdom Patent Specification 843,849. The melt compounded mixture of polymethyl methacrylate and barium sulphate as described in Example 1 was injected as a melt into the bypass by means of a ⅝" single screw extruder. The main extruder was fitted with a 4' wide sheet die. The rate of pigment injected could be easily varied to give sheet with varying light transmission characteristics and excellent pigment dispersion. An ⅛" thick sheet had a 55% light transmission when the pigment compound was added at 8 lb./hr.

WHAT WE CLAIM IS:—

1. An extrusion process in which a molten mass of a thermoplastic polymeric material (A) is extruded utilising screw extrusion apparatus having at least two zones through which the polymeric material (A) is conveyed by means of a screw or screws and a duct connecting said zones so that the thermoplastic polymeric material (A) can pass from one zone to the other, characterised in that an intimate mixture of an additive and a thermoplastic polymeric material (B) is injected into said duct in the form of a melt while the molten thermoplastic polymeric material (A) is passing therethrough.

2. An extrusion process as claimed in Claim 1 in which the mixture of the additive and the polymeric material (B) consists of a masterbatch prepared by melt compounding the polymeric material (B) with the additive.

3. An extrusion process as claimed in Claim 1 or Claim 2 in which the thermoplastic polymeric materials (A) and (B) are the same.

4. An extrusion process as claimed in any of Claims 1 to 3 in which the thermoplastic polymeric material (A) is polymethyl methacrylate.

5. An extrusion process as claimed in any of Claims 1 to 4 in which the thermoplastic polymeric material (A) is extruded through at least two separate screw extruders one feeding directly into the feed point of the other via a short unstirred duct into which duct the mixture of the additive and the polymeric material (B) is injected.

6. An extrusion process as claimed in Claim 5 wherein the thermoplastic polymeric material (A) is extruded through a twin screw extruder into the barrel of a single screw extruder through a short unstirred duct into which duct the mixture of the additive and the polymeric material (B) is injected.

7. An extrusion process as claimed in any of Claims 1 to 4 in which the thermoplastic polymeric material (A) is extruded through an extruder having two zones separated by a cylindrical member fastened to the extruder screw and rotatable therewith which member acts as a 'plug' to prevent flow of the polymeric material (A) along the extruder barrel from one zone to the other and having a duct passing along the barrel wall connecting the zones thereby bypassing the cylindrical member characterised in that the mixture of the additive and the thermoplastic polymeric material (B) is injected into said duct.

8. An extrusion process as claimed in any of Claims 1 to 7 wherein the mixture of the additive and the thermoplastic polymeric material (B) is injected into the duct by a screw extruder.

9. An extrusion process as claimed in any of Claims 1 to 8 in which the molten mixture of the thermoplastic polymeric material (B) and the additive is injected into the duct in a direction counter to the flow of the polymeric material (A) through the duct.

10. An extrusion process as claimed in any of Claims 1 to 9 substantially as hereinbefore described with particular reference to the examples.

C. GRATWICK,
Agent for the Applicants.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1971.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

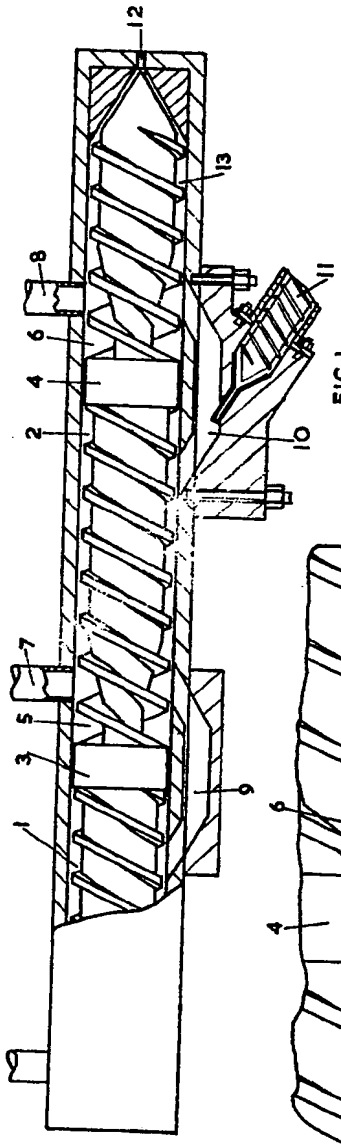

FIG. 1.

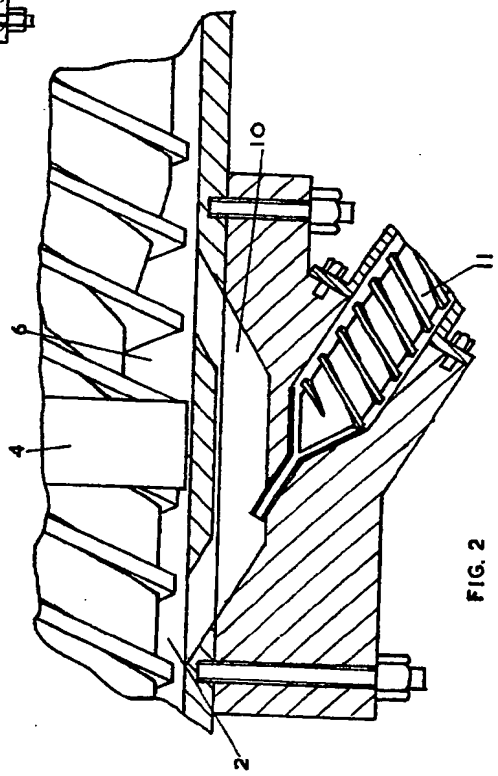

FIG. 2